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STUDY ON WAX DEPOSITION OF HEAVY CRUDE OIL IN PIPELINE
TRANSPORTATION BY USING OIL IN WATER EMULSION METHOD

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ABSTRACT

Oil in water emulsion occurs in conventional crude oil transportation process. The emulsion stability results from the high concentration of asphaltene, wax, resins and naphthenic acid on the composition of emulsion will causing the appearances of wax deposited on the wall of the pipelines, this condition increasing the viscosity of the crude oil emulsion and the pipeline damage and causing problem to the pipeline transportation. In this study the oil in water emulsion was prepared by mixing of the different volume ratio of oil and water. By using the different concentration of surfactant, different speed of mixing and different temperature of mixing, the stability of the condition will be tested. Instead of stability and the viscosity, the shear rate, shear stress, and its density were determined by Brookfield viscometer. The demulsification is also being applied on this study to study the best demulsifier to break the emulsion. It is expected that, the most stable emulsion is that with higher concentration of surfactant, highest speed and the higher volume of the oil. By increasing the mixing speed, lowering temperature and increasing time of speed results increasing the viscosity of the emulsions.

MENGAJI ENAPAN LILIN MINYAK MENTAH DALAM PENGANGKUTAN TALIAN PAIP DENGAN MENGGUNAKAN KAEDAH EMULSI MINYAK DALAM AIR

ABSTRAK

Emulsi minyak dalam air yang berlaku dalam proses penyangkutan konvensional minyak mentah. Kestabilan emulsi dari kepekatan asphaltene, bahan berlilin, resin dan asid naftenic pada komposisi emulsi akan menyebabkan kewujudan lilin yang terlekat di dinding saluran paip. Keadaan ini akan meningkatkan kelikatan emulsi minyak mentah dan saluran paip akan menyebabkan masalah kepada pengangkutan saluran paip. Dalam kajian ini, minyak dalam air emulsi telah disediakan dengan campuran nisbah isipadu minyak dan air yang berbeza. Dengan menggunakan kepekatan surfaktan, kelajuan dan suhu yang berbeza, keadaan akan diuji dan dianalisa. Selain kestabilan dan kelikatan, kadar ricih, tegasan ricih dan ketumpatan ditentukan dengan menggunakan meter kelikatan Brookfield. Menyahmulsi juga dikaji untuk mengkaji penyahmulsi yang terbaik untuk memecahkan emulsi. Dalam jangkaan, emulsi yang paling stabil adalah emulsi dengan kepekatan yang mempunyai kepekatan surfaktan yang tinggi, kelajuan yang tertinggi, suhu yang rendah dan isipadu minyak yang tinggi akan meningkatkan kelikatan emulsi.

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LIST OF SYMBOLS

°C	Degree Celsius
°K	Degree Kelvin
RPM	Rotation per minutes
mL	Mililiter
%	percentage

LIST OF ABBREVIATIONS

HLB	- Hydrophilic-Lipophilic balance
HMWH	- Higher molecular weight hydrocarbon
O/W	- Oil in water emulsion
PPD	- Pour point depressant
SARA	- Saturates(including waxes), aromatics, resins, asphaltene.
Span-80	- Sorbitan Monooleate
Span-83	- Sorbitan Sesquioleate
Triton X-100	-Polyethylene Glycol Octylphenol Ethoxylate
LSWR	- Low Sulphar Wax Residue
W/O	- Water in oil emulsion
WAT	- Wax Appearance Temperature

CHAPTER 1

INTRODUCTION

1.1 Research Background.

Heavy crude oil is in term of ‘unprocessed’ oil also known as Petroleum is the mixture of naturally organic compounds which is contains primarily hydrogen, carbon and oxygen. Crude oil is a complex mixture of fluid comprising colloid particle such as asphaltene and resins aggregates. These particles are dispersing in a mixture of aliphatic and aromatic solvent (Langevin *et al.*, 2004). Initially, heavy crude oil is a dark and viscous liquid with its condensate is a clear and volatile liquid. Hence, crude oil usually in black color but also comes in other uncommon colors likes green, red, or brown. The heavy crude oil consists of hydrocarbon chains in their chemical structure at various length, as known, hydrogen and carbon are two main atomic involved in hydrocarbon chain which are come in straight branching to rings. Crude oil can be characterized by the so-called SARA (saturated, aromatic, resins, asphaltenes).

Heavy crude oil eventually contains large quantities of asphaltenes which is high molecular weight polar component that react as natural emulsifier. The other surface active are resins, fatty acid including naphthenic acids porphyrins and wax crystal, but, most of the time they cannot produce stable emulsion alone (Langevin *et al.*, 2004). Presence of asphaltenes may produce stable emulsion. In this case, emulsion with combined particle can be much more stable than those stabilize by asphaltene alone, all the particle need to be saturated by asphaltenes to ensure stable emulsion.

Knowledge of emulsion is crucial for improving and controlling the process of oil transportation process. Emulsion of crude oil and water can be staged at many stages during drilling producing, transportation and processing of crude oil in various including hydrocarbon reservoir, well bores, surface facilities, transportation and refineries. Water in oil emulsion is formed during the production of crude oil, which is often accompanied with water. The emulsion is stable in ranging from a few minutes to years depending on the nature of the crude oil to extend the nature of water. The secondary production of natural petroleum includes of crude oil as dispersion medium and salty water known as brine as dispersed phase, it is naturally stabilized by natural emulsifier such as asphaltene, resins, solid such as clays and waxes (Bhardwaj, 1998).

As known, crude oil consists of a series of hydrocarbons such as alkanes, naphthalenes, and aromatic compound such as phenols, metals and carboxylic acids. As well, a major fraction of nitrogen and sulfur compounds present. The carbon numbers of these entire component range from carbon-1 (methane) through 50 or

more as example asphaltene. According to Schramm (1992) has stated that some of these components can form films at the oil surfaces and other active surface. So, the tendency to form stable or unstable emulsion of variety is various among different oil.

Asphaltenes is react as surfactant as the larger molecular compound and require longer adsorption, and the adsorption is irreversible in water oil emulsion(w/o), as surface active substances in asphaltene, it is spontaneously adsorb at the surface. So, the surface tension is low (Langevin *et al.*, 2004). In case of small surfactant molecule, a monolayer of monolayer are formed, with polar part of the surface active molecule in contact with water, hence, hydrophobic part is also contact with water. Comparison between crude oil model emulsion system require that, resins and asphaltenes fraction are indicates for the effectiveness of solid particle in crude oil emulsion stabilization. So, extend of asphaltene aggregation is the crucial and major factor for controlling solid stabilization effectiveness. It is shows that, water-oil emulsion are extremely stable because of the presence of asphaltene and resins. The natural surfactant adsorbed on the surface of water droplets and form physical barrier which means the interfacial film with viscoelastic properties. This condition will prevent coalescence of water droplet (Filho *et al.*, 2012). To reduce water content or broke it of produced crude oil, the water with crude oil emulsion need to be demulsified; hence it is important to understand the mechanism for stabilization emulsion.

Several techniques are being applied to treat of water-crude oil emulsion and oil in water emulsion which involves the application of chemical, thermal and

electrical process of combination. Chemical methods are the most common used in both oil field and refinery. The combination of chemical and heat designed to stabilize the effect of emulsifying agent have great advantage of being able to break on interfacial film effectively, which is without the addition of new equipments or modify existing equipment.

Thermal method or known as heat treatment in demulsification is basically on the overall economic picture of a treating facility. Excess heat is not important when it is more efficient to add chemical or set up electrostatic heat. High temperature does not cause asphaltene to become insoluble in the crude oil. Electrical methods interfere the surface tension of each droplet, maybe by causing polar molecule to reorient themselves, this situation is weakens the film around each droplet

1.2 Problem Statement.

Presence of wax deposition in crude oil has caused flow and mechanical problems to the pipeline transportation, also in production and storage of crude oil, this situation occurs because the oil temperature drops along the pipeline, hence, the quantities of wax crystal increase to be formed in the crude oil phase. This condition is much high viscosity level, then the higher pumping power expressed in shear effect needed to easy pipeline transportation. (El Gamal, 1998). The wax crystal usually lead to higher viscosity with increased energy consumption for pumping and decreased capacity, the wax crystal is cooled during transportation. Wax deposits may cause to increased roughness of the pipeline hence reduced its diameter

effectively, this problem potentially required frequent pigging and causing the problem to pigging if the wax deposits is too thick. It is costly to recover these pipeline problems.

The understanding of the emulsion stabilization is crucial for controlling the viscosity of crude oil transportation process. In order to minimize the production and transportation of crude oil and environmental concern, formation of emulsion need to recover to increasing the flow enhancement.

1.3 Research Objectives.

At the end of this study it is necessary to:

- To evaluate the effect of asphaltene and molecular weight of flow improver on the rheological behaviors of heavy crude oil.
- Preparation of model emulsion and their characteristic by study the stability using different type of surfactant with different concentration, different volume ratio oil in water emulsion and its chemical demulsification.

1.4 Scope of Research.

This research is to study wax deposition of heavy crude oil in pipeline transportation, thus to achieve the objective the characterization of crude oil in terms

of physic-chemical properties should be known and be investigated to get the information regarding the crude oil properties. Moreover, the hydrocarbon chain distribution also needs to be investigating to determine the changes of characteristics in crude oil according to various hydrocarbon chains, in this study, the hydrocarbon chain distribution is determined by using different volume of oil and water which is by using 50-50 %, 40-60 %, and 30-70 % of oil in water emulsion. Surfactant is the component that encourage the stable emulsion on crude of hence this research will study the type of surfactant and its concentration on crude oil, this study will use different types of surfactant with different concentration of 0.5 %, 1.0 % and 1.5 %. Meanwhile, the viscosity and the temperature dependence also will be investigated on crude oil transportation process, in this study, different mixing speed is be used which is 500 rpm, 1000 rpm, 1500 rpm and 2000 rpm with time of agitation 10 minutes.

1.5 Significance of Study

By study of the wax deposition on pipeline system, it is expected to study factor and the way to overcome this problem that naturally will give the problem to crude oil processing and transportation pipeline. The viscosity reduction is the main idea of study of this research, hence, by creating the emulsion model and determines the most stable condition of emulsion stabilization of the oil in water emulsion. Then the effective way to break it is to investigate the efficient method and to reducing the problem related to transportation in pipeline system related to wax deposition on pipeline.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction.

Heavy crude oil also known as petroleum is the stuff that comes out of the ground, especially contains large quantities of asphaltenes with high molecular weight polar component that acts as natural emulsifier. Crude oils are a continuous fluid contain of tens of thousands of different hydrocarbon molecule. Sjöblom (2003) shows the proportions of the elements in crude oils have large variation in properties from the lighter crude oil to heavy asphaltenic crudes. Normally, the carbon content is in the range 83 to 87 percents hence, the hydrogen content varied of 10 and 14 percents, Due to the crude oil complex composition, characterization by the individual molecular type is not possible. Hence, the SARA-separation is the hydrocarbon group type analysis that is commonly employed, Figure 2.1 shows SARA separation scheme, SARA-separation is such group type analysis which separating the crude oil in four main chemical classes based on differences in solubility and polarity. Crude oil is a complex fluid comprising colloidal particle

such as asphaltenes and resins dispersed in a mixture of aliphatic and aromatic solvent (Langevin, 2004 & Kumar, 2001).

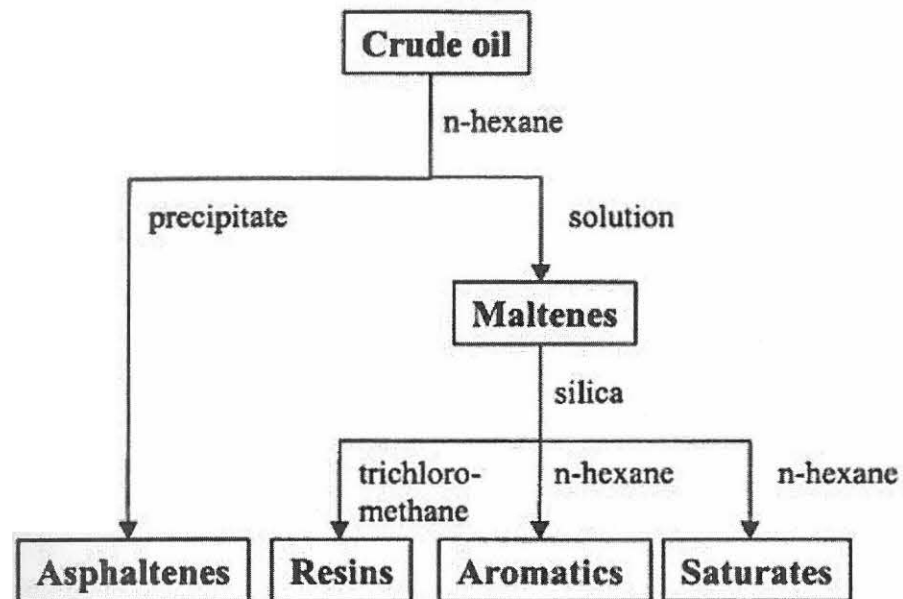


Figure 2.1: SARA separation scheme (Sjöblom, 2003).

Crude oil is the fossil fuel, which is made up naturally from decaying plant and animal living in ancient seas, millions years ago, crude oil is vary in color of from clear to black. Crude oil contain hydrocarbon molecule that contain hydrogen and carbon with various length and structure. The crude oil particularly content high molecular weight paraffins and asphaltenes have significant impact related to deposition of waxes during transportation and production. Vara (2011) previous studies have shown that oils with wax composition with at least 2 % have the potential for wax deposition problems (Thanh *et al.*, 1999). As we know, crude oil is a mixture of light and heavy hydrocarbons, the heavier part which is wax and asphaltene are dissolves in the light crude oil, when the temperature drops at given pressure, the solubility of heavy fraction will deduce and the heavy hydrocarbon

precipitated will start forming of wax and asphaltene, further of the temperature decrease, the amount of solid precipitated from crude oil will decrease (Chen *et al.*, 2007).

Many problems related to wax, asphaltene, hydrates which may lead to deposition causing pressure drop, reducing production and risk of pipes plugging. Hence, slugging also one of the problems occurs as elevated viscosity and gelatin at low temperature operating system represent this potential rheological problem (Mohammed, 2010). High content of wax could be disadvantageous in crude oil as well as bitumens. In crude oil transportation, wax may crystallize at low temperature and precipitate as a solid material causing problems in pipelines and in production and processing equipment (Lu *et al.*, 2008). Garcia *et al.*, (2000) purport that paraffin deposition down hole and in surface equipment is one of the most serious problems in oil production operation, there will makes changes in physico-chemical equilibrium according to decreasing in temperature below paraffin melting point cause crystallization, losses in component solubility and sequence of accumulation.

Moreover, precipitation of wax increase significantly due to temperature decrease, this transition typically occurs about 10-15 °C below the Wax Appearance Temperature (WAT) : Kok *et al.*, (1996) stated that, WAT is the temperature at which visible crystallization occur, it is depending on the molecular weight and the concentration of the waxes and the chemical nature of non-waxy part of crude oil in term of hydrocarbon. According to Vara (2011), the lowest temperature of crude oil can flow under static condition is known as pour point temperature (PPT) : Kok *et.al.*, (1996) stated that, PPT as the temperature falls, crystal continuous and

simultaneously the amount waxes precipitated increases. Pour point usually determined by using the ASTM D97-87 procedure.

2.2 Crude oil Emulsion Composition

2.2.1 Introduction

Crude oil contains complex mixture of organic composite. Its composition can vary due to its reservoir's place of origin, depth and age. Hydrogen and carbons are the mixture that's mainly consist on the crude oils, while, little amount of sulphur, nitrogen and oxygen as well as structure with incorporated metallic molecules such as nickel, vanadium, copper and iron (Speight, 1991). There is a ample properties from the lighter oils to the bitumen. For this reasons, several classification systems of petroleum were proposed based on different criteria: viscosity, density, (specific gravity or API gravity), pH, surface tension and interfacial tension.

The fraction of crude oil that have been identified as contributing to the formation of oil in water emulsion includes asphaltenes, resins and waxes and can exist in both the dissolved and particulate form (Lee, 1999).

The basis method to remove asphaltenes is by precipitation in paraffinic solvent such as n-pentane. Chromatographic fractionation method is used to separate the deasphalted oil into saturated, aromatics and resins (Aske *et al.*, 2001). From the

four classes of compounds, only the saturated are easily discernible from the rest of the hydrocarbon in the mixture. This is due to the absence of π -bonds, which allows them to be readily differentiated from the aromatic component by asset of the difference in their polarity. The balance of the oil is contained aromatics and heteroatomic compounds of varying degree of functionalism, alkyl substitution and condensation.

The saturated or aliphatics are non-polar hydrocarbons, having branched alkanes and straight-chain but without double bonds, as well as cycloalkanes or naphtenes. Cycloalkanes contain one or more rings, which may have several alkyl side chains. The proportion of saturates in a crude oil normally decrease with increasing molecular weight fractions, Thus the saturated generally are the lightest fraction of the crude oil. Wax is a sub-class of saturated, consisting primarily of straight chain alkane. Wax precipitates as a particulate solid at low temperature and is known to effect emulsion stability properties of crude oil systems (Zaki *et al.*, 2000).

Aromatics are common to all petroleum, these terms refer to benzene and its structural derivates and by far the majority of the aromatics contain alkyl chains and cycloalkane rings, along with additional aromatic rings. Aromatics are often classified as mono-, di- and tri- aromatics depending on the number of aromatics ring present in the molecule. Polar, higher molecular weight aromatics may fall in the resin or asphaltenes fraction (Aske, 2002). The term of asphaltenes and resin will be discussed in section 2.2.2 and 2.2.3 respectively.

2.2.2 Asphaltenes

Asphaltenes are dark brown to black amorphous powder and have a specific gravity just above unity, and molar masses of 1000 to 10,000 g/mol (Speight, 1994). Asphaltenes has no definite melting point but decomposes when the temperature exceeds 300-400 °C. Many researches had shown that changing in pressure, temperature and oil composition cause asphaltene precipitation.

Asphaltene are the non volatile and polar fraction of petroleum that is insoluble in n-alkanes such as n-pentane or n-heptane. So, asphaltenes represent of crude oil component, rather than a chemical class. The polarity, molecular weight and aromaticity of precipitated asphaltenes are rise linearly with carbon number of n-alkane precipitate.

The chemical compositions of crude oils, gained from for instance a SARA analysis are not fully explaining the crude behavior with regard to emulsion stability and asphaltene deposition. The information of the structure of the crude oil which is a result of interaction between the continuous of chemical constituents in the oil is the most important. The interaction between the heavy and molecules, the asphaltenes and resins. play the most significant role in this sense.

Asphaltenes are interfacial active substance exist predominantly in the crude oil colloids. The interfacial active component of the asphaltenes are most active either directly before or during the start of flocculation (Schorling *et al.*, 1998). Asphaltenes also contain metal including nickel, vanadium and iron.

The structure of asphaltenes is not well understood, but several possible structures have been proposed to explain the composition and properties of the asphaltenes fraction. The structure of asphaltenes which account for nitrogen, sulphur, and oxygen are excluding the organometallic. An organometallic complex is also shown which is assumed to complex other asphaltene compounds in the micelle. Such asphaltene sheets appear to be regularly stacked in lamellar and such structure, which are found in other surfactant system, are known to enhance the emulsion stability (Lee, 1999).

2.2.3 Resin

Resins are black or dark brown semi solid, have a specific gravity near unity, molar mass ranging from 500 to 2000 g/mol and very adhesive materials (Gafonava, 2000). The content of resin in crude oils ranges from 2-40 %, the content of resins in crude oil is higher compared to asphaltenes composition. Crude oil with a small amount or no asphaltenes has a lower concentration of resins than those with large amount asphaltenes.

The main Fraction consists of carbon, hydrogen, oxygen, nitrogen and naphthenic acids, the content of these elements in resin of various over a narrow range. The widest range is observed in sulfur content. Resins have a much higher hydrocarbon ratio compared to asphaltenes, indicating that they are less aromatic than asphaltenes, Asphaltenes are presumed to be maturation products of resin, in the